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## DEFLAGRATION-TO-DETONATION TRANSITION IN LX-04 AS A FUNCTION OF LOADING DENSITY, TEMPERATURE, AND CONFINEMENT

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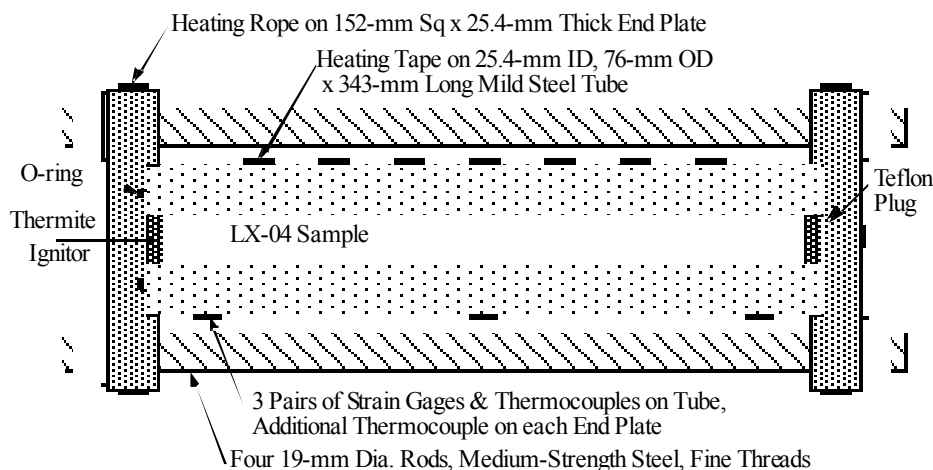
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**Abstract.** The potential for deflagration-to-detonation transition (DDT) in LX-04 (85/15 HMX/Viton) is being evaluated as a function of loading density, temperature, and confinement. In the high confinement arrangement, a matrix of tests is nearly completed with the LX-04 loaded at ~51, 70, 90, and ~99% of theoretical maximum density (TMD); and temperatures of ambient, 160°C, and 190°C at each loading density. A more limited set of tests with ~99 %TMD loadings at medium confinement were conducted at temperatures of ambient and 186°C. LX-04 does not undergo DDT at near TMD loadings in both medium and high confinement, although the latter still results in significant fragmentation. Most porous beds in high confinement undergo DDT, with the minimum run distance to detonation (*l*) for a 70 %TMD loading at ambient temperature. LX-04 does not transit to detonation for a pour density (51.3 %TMD) loading at 160°C, but does at 190°C with a longer *l* than at ambient. The limited ambient temperature measurements for *l* in high confinement are similar to previous data for 91/9 HMX/wax, which has nearly the same %volume of HMX as LX-04.

### INTRODUCTION

The objective is to determine if a violent event could occur from accidents in which LX-04 is both damaged and subjected to fire. Violence is based on fragmentation of the confining apparatus, the extreme being from sample detonation. The potential for detonation is the run distance from the ignitor to detonation (*l*), with short *l* having the highest potential. Since the LX-04 may be damaged, the loading densities vary from a loose bed of molding powder, which is ~51% of theoretical maximum density (TMD), to

intermediate loadings at 70 and 90 %TMD, to highly pressed samples at ~99 %TMD. The test matrix includes temperatures of ambient, 160°C (just below the HMX phase transition), and 190°C (just above the HMX phase transition). All combinations of density and temperature will be tested in high confinement, which is greater than could ever exist in an accident, to determine any potential for DDT. Some tests for near TMD loadings were conducted at a more realistic, medium confinement.



**FIGURE 1. High confinement apparatus.**

## TEST ARRANGEMENTS

The high-confinement apparatus shown in Figure 1 is mostly that used by Bernecker et al.<sup>1</sup> in which the confinement tube, end plates, and threaded rods were all of mild steel. The ignitor end of the tube is sealed with a Viton O-ring in the end plate, whereas the other end of the tube is not sealed and would permit venting during heating. End confinement was somewhat increased with rods of a higher strength Grade B7 alloy steel having fine instead of coarse threads to increase their cross-sectional area. For withstanding the high temperatures in some of the present tests, the ignitor header and its leads were made from Teflon. When installing the ignitor, the leads were twisted together and bonded into a hole through the end plate with an epoxy having integrity at 190°C. The Micro-Measurements M-Bond AE-15 epoxy was also used to bond strain gages (SGs) and thermocouples to the apparatus. Even though the circumferential strain in a thick-wall tube at failure may be only several percent, high elongation SGs of annealed constantan were selected so that the same ones could be used on the medium-confinement apparatus. An interesting deviation from the previous arrangement was the use of a 0.65 g thermite ignitor, which only generates heat instead of the hot particles and gas from the usual  $\frac{3}{4}$  g B/KNO<sub>3</sub> ignitor, so that onset of LX-04 reaction is more like that in thermal explosion. Most heated tests on compacted beds had some ullage adjacent to the ignitor to accommodate bed expansion.

Different loading techniques were required for varying the LX-04 density. All porous beds were loaded in 19-mm high increments by pouring the molding powder for each increment into the tube and pressing. Each increment in a loose bed was hand pressed with insignificant compaction mostly to verify a consistent height. The 70 and 90 %TMD beds were remotely pressed into tubes shortened to 314.3 mm to accommodate the pressing hardware. Because of the high binder content in LX-04, it was necessary to over-press 70 %TMD beds by ~2 mm and 90 %TMD beds by ~1 mm. These increments relaxed to 19 mm within the several minutes before the next increment could be loaded. All tubes loaded with porous beds had a 9.5-mm high Teflon plug in the far end, which was necessary for compacted beds to prevent relaxation of the LX-04 from the end of the tube during assembly of the apparatus. In time, the compacted LX-04 beds would stress relax and no longer expand when unrestrained. Since the mild steel tubes are not strong enough for pressing increments at full density, ~99 %TMD, these tubes were sized so that light pressure was required to insert 25.4-mm high pellets machined at PANTEX from isostatically pressed LX-04. For the test at ambient temperature, about 60 mg of vacuum grease was on the downstream end of each pellet, which was pressed down with a ram by hand to force some of the grease to the inner tube wall to inhibit gas flow along it. As pressure in the ignitor region would begin to increase during a test, more grease will be forced to the wall until pressures are high enough to



Insulated

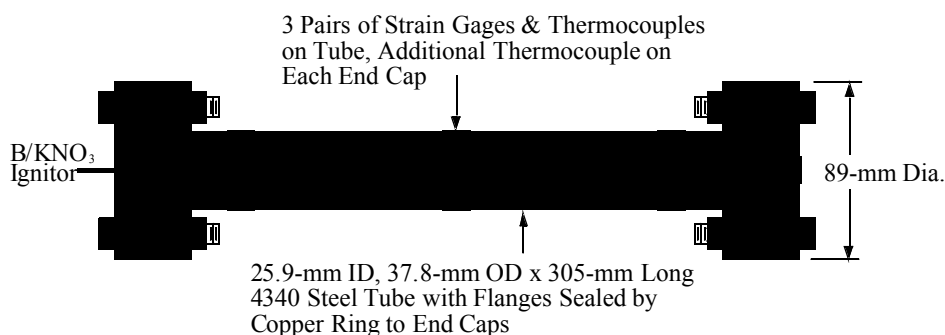
**FIGURE 2. An assembled high-confinement apparatus for a heated test before and after insulation.**

deform the pellets against the wall. For heated tests, the greater thermal expansion of LX-04 than steel would seal the pellets to the inner wall.

Each end plate and the tube had separate heating elements with the power to them individually controlled, but all were cycled on and off at the same time. With this heating arrangement and the wrapping of the entire apparatus in R13 insulation, the temperature over it varied less than 2°C. Photographs of an apparatus with the heating elements installed before and after insulation are shown in Figure 2.

The medium-confinement apparatus shown in Figure 3 is an adaptation of the design for the STEX test<sup>2</sup> at the Lawrence Livermore National Laboratory (LLNL) in which the tube is brazed to flanges and each end is closed by a cap with a copper ring for sealing. The tube with its flanges and the end caps are 4340 steel hardened to Rc 32, and each end cap is secured to the flange with eight 9.5-mm diameter high-strength bolts. The tube has a 6.0-mm thick wall versus the 25.1-mm thick wall in high confinement arrangement. The thinner wall

of hardened steel provides adequate strength for the slow pressure buildup during the early stages of DDT, but has less inertial confinement than the thicker wall in the high confinement apparatus during the rapid pressure buildup in the final stages of DDT. The only two tests conducted to date with this arrangement were at LLNL, one at ambient temperature and the other at 186°C, which was obtained with the apparatus in a convection oven. These tests had ~0.45 g of B/KNO<sub>3</sub> in the ignitor and were loaded with 25.3-mm diameter LX-04 pellets that had been hydraulically pressed to ~99 %TMD. Because of the 0.3-mm annulus between the pellets and the inner tube wall, a measured amount of an RTV potting compound was added before inserting each pellet. Some pressure was applied to the pellet to force the RTV into the annulus around it. A 23.5-mm long section beyond the ignitor was not loaded with LX-04 for the heated test, corresponding to 8% ullage for accommodating the HMX expansion during its phase change. Only the heated test had a single SG mounted at 41.3 mm from the ignitor interface.



**FIGURE 3. Medium confinement apparatus.**

## TEST RESULTS

Summaries of the completed tests are listed in Table 1. The high-confinement tests at pour density were dependent on temperature, with DDT at ambient temperature and 190°C, but not when heated to 160°C. At 190°C,  $l$  was somewhat longer than at ambient temperature. A similar trend occurred at 70 %TMD, except that  $l$  was much less so that detonation occurred near the end of the bed in the 160°C test. In the only test to date at 90 %TMD,  $l$  was slightly greater at ambient temperature than for the pour density bed at that temperature. The tube in this test had only a 228.6-mm long bed followed by a 76.2-mm long Teflon plug to avoid exceeding a 200-g limit in the pressing bay.

DDT did not occur with LX-04 at near TMD for either confinement at both ambient temperature and 190°C. A high confinement test with a minimal 1% ullage adjacent to the ignitor was planned for 160°C, but ignition failed. (In other tests, igniting with thermite had delays of ~60 ms and sometimes >100 ms, versus 0.5 to 3.5 ms<sup>3</sup> with a ¾-g B/KNO<sub>3</sub>

ignitor, and may have been marginal without the pressurization from some gas products in B/KNO<sub>3</sub>.) The heating of this apparatus was continued at 0.7°C/min until cook-off occurred at 224°C; and so the test is tabulated in the 190°C column. In the medium-confinement test heated to 186°C, the 8% ullage was completely filled by the phase change in HMX and thermal expansion of the composition. This overall reduction in density did not increase the potential for DDT as indicated by the bulged tube versus the broken one at ambient temperature.

Recovered apparatus from the medium-confinement tests had the ends still closed with splits in the tube. The end closures in the high-confinement apparatus were not nearly as strong as the tube, and the downstream end plate was often punched through, even in tests without DDT. In the test at pour density and 160°C, the downstream end of that tube had bulged, consistent with the damage to its end plate. This localized tube deformation was from rapid pressurization that may have achieved DDT in a longer tube. Of the two tubes loaded with pellets at near TMD, the test at ambient

**TABLE 1. Summary of Tests**

Confinement	LX-04 Density (%TMD)	Reaction Violence versus Temperature		
		Ambient	160°C	190°C
High	51.3 <sub>avg</sub>	$l = 194$ mm	Bulged tube	$l = 267$ mm
	70	$l = \sim 114$ mm	$l = 263$ mm	$l = 190$ mm
	90	$l = \sim 204$ mm		
	98.6	~20 tube fragments		Cook-off without tube deformation at 224°C
Medium	98.6	3 tube fragments		Bulged tube

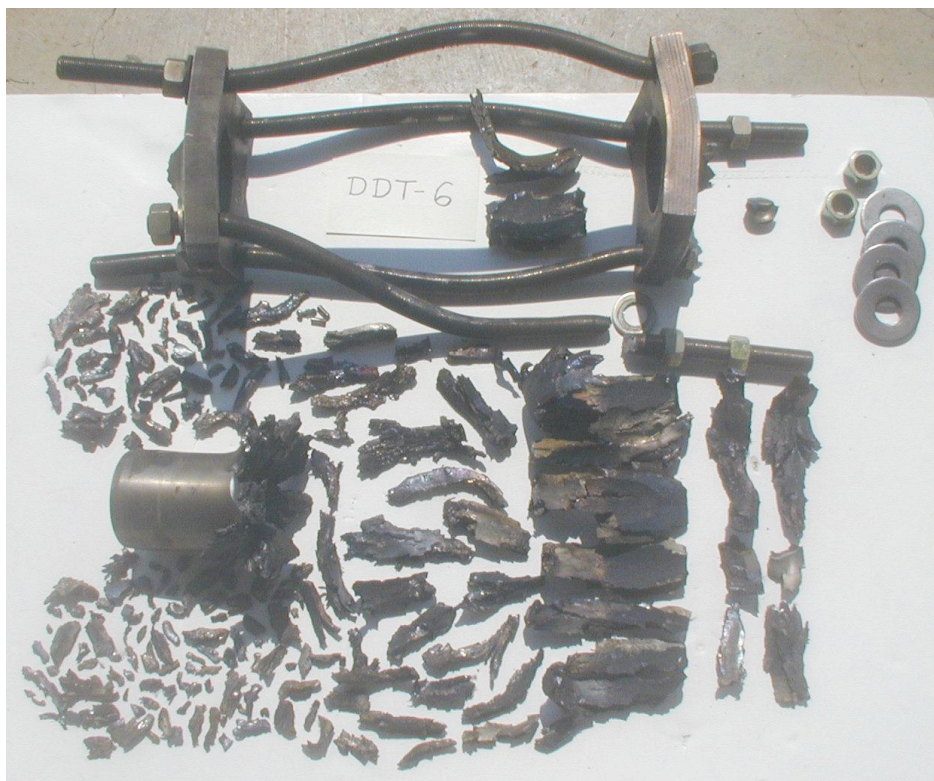
$l$  = run distance to detonation from the ignitor

temperature had the greater damage to the apparatus; the tube was somewhat uniformly fragmented into ~20 pieces and both end plates were punched through. For the apparatus that cooked off, the tube was not even expanded and the end plates were only deformed.

When DDT occurred in the other high-confinement tests, both end plates were punched and the tube was extensively fragmented. The onset of detonation is evidenced by a distinct boundary with micro-cracking and blackening on the inner wall of the tube. Those characteristics persist beyond the onset of detonation, where the tube is broken into <10 long fragments. The ignitor end of the tube was intact in the two pour density tests with DDT and in the tube loaded at 90 %TMD with a reduced bed length; however, the ignitor end was broken into large pieces for the tests at 70 %TMD. Between the ignitor region and the onset of detonation, the tubes were shattered into small pieces without the characteristic micro-cracking from detonation. This is illustrated in Figure 4 for

the pour density test at 190°C, where the ignitor end is on the left side in the photograph and only large fragments are approximately located; the many small fragments above and below the ignitor section came from the middle region of the tube. The extensive damage, which increased with loading density, made a measurement of  $\ell$  difficult, requiring the SG measurements for guidance.

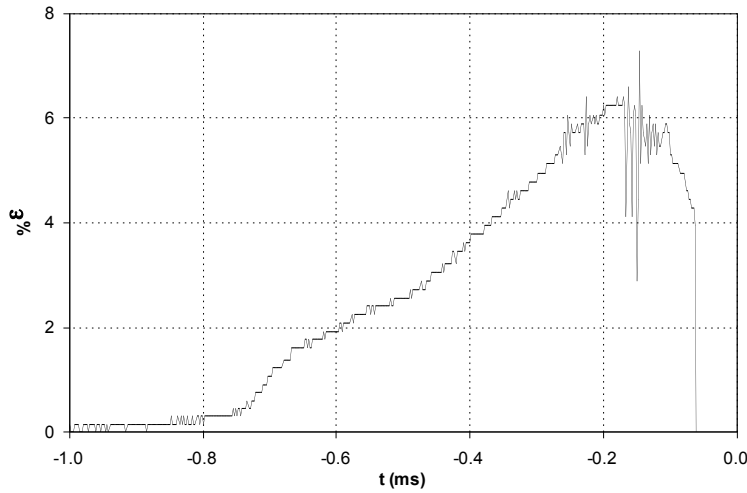
Strain-time ( $\epsilon$ - $t$ ) traces for tests that bulge the tube versus undergoing DDT are contrasted in Figures 5 and 6. The sensitivity of SGs for elastic tube expansion is 1.05 % $\epsilon$ /GPa for the medium-confinement tube versus 0.25 % $\epsilon$ /GPa for the high-confinement tube. Elastic expansion ends with yielding at the inner wall for the medium-confinement tube at 0.27 GPa or 0.28 % $\epsilon$ , versus at 0.18 GPa or 0.045 % $\epsilon$  for the high-confinement tube; thus the displayed traces are mostly plastic deformation at high strain rates and would require modeling to relate to interior pressure. For the medium confinement tube loaded at near TMD and



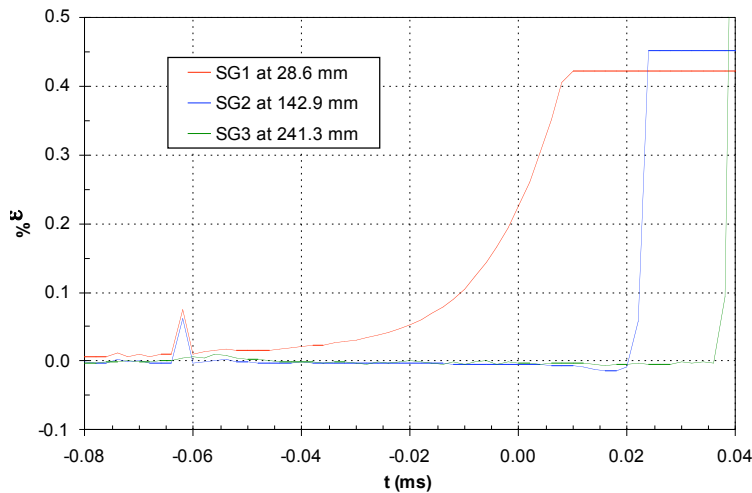
**FIGURE 4. Recovered high-confinement apparatus from test at pour density and heated to 190°C.**

heated to 190°C, the single SG trace in Figure 5 shows a relatively linear increase in tube expansion for more than 6 % $\epsilon$  until either the tube has split and released the gas products or a SG lead connection failed. For the high confinement tube loaded at 70 %TMD and not heated, the traces in Figure 6 show the development of DDT. The trace of the SG nearest the ignitor increased exponentially in an order of magnitude shorter time than in the previous figure. The next SGs are beyond 1 and have essentially a step increase in signal with the arrival of the detonation wave, whose average velocity is 5.5 mm/ $\mu$ s. Peak values of  $\epsilon$  shown in Figure 6 were limited by oscilloscope settings and were actually several percent on

another oscilloscope with less resolution. When the same loading is heated to 160°C, the traces in Figure 7 (same  $\epsilon$  scale as Figure 6) show a different behavior. Near the ignitor,  $\epsilon$  increased almost linearly to a plateau of 0.054%, corresponding to only  $\sim$ 0.22 GPa; and a similar linear increase occurred at the center of the tube. The SG near the end of the tube recorded a rise almost as steep as the last two SGs in the previous figure, indicating it was very near the onset of detonation, consistent with tube fragments. After the rapid increase in  $\epsilon$  near the end of the tube, the SGs near the center of the tube and the ignitor respond to a rearward  $\sim$ 7 mm/ $\mu$ s wave.

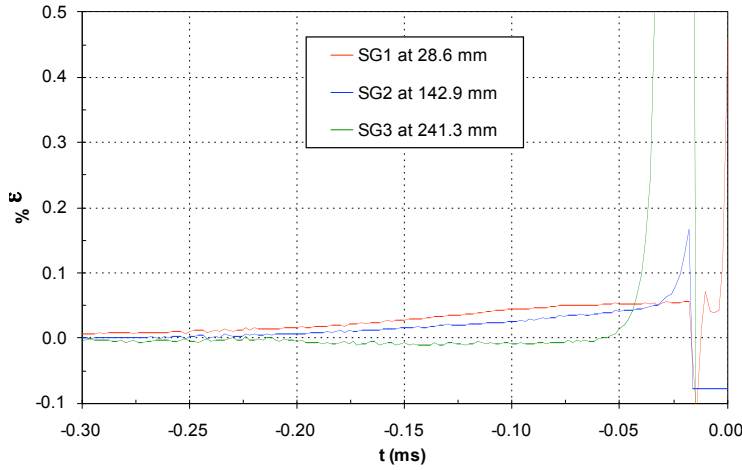


**FIGURE 5. Strain at 41.3 mm on medium-confinement tube loaded at near TMD and heated to 186°C.**



**FIGURE 6. Strain for high-confinement tube loaded at 70 %TMD and not heated.**



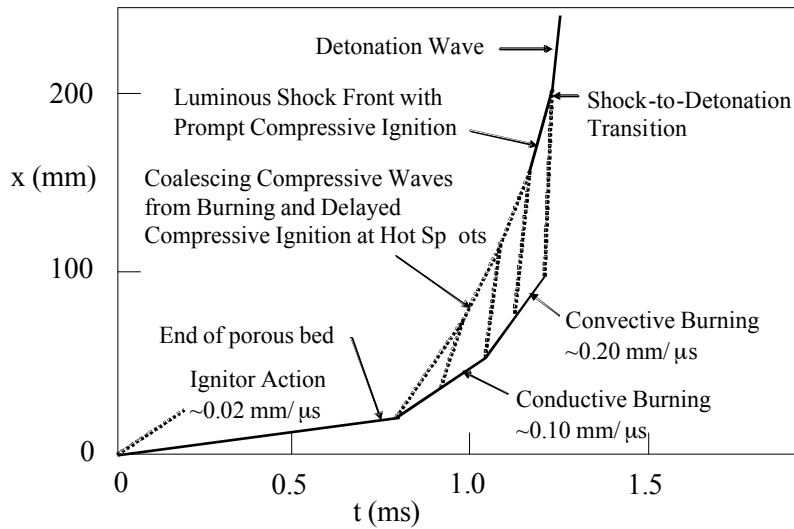


**FIGURE 7. Strain for high-confinement tube loaded at 70 %TMD and heated to 160 °C.**

## DISCUSSION

The reduced density from heating near-TMD LX-04 beyond the temperature for the phase change in HMX did not result in an any inner-connected porosity for accelerated burning, as indicated in Figure 5 by the nearly linear increase in  $\epsilon$  occurring over  $\frac{1}{2}$  ms. Without a rapidly accelerating pressure buildup from convective burning to drive a strong compressive wave for initiating compressive reaction, which will then drive a shock wave, there was no opportunity for shock-to-detonation transition (SDT), the last step for DDT<sup>4</sup> as

illustrated in Figure 8. This is in contrast to the accelerating tube expansion near the ignitor that occurs in an order of magnitude less time, as shown in Figure 6 for a 70 %TMD bed. Heating such a porous bed to 160°C, however, reduced the early pressurization rate because the softened Viton binder compacted more easily, thereby inhibiting convective burning. The  $\epsilon$  plateau at the ignitor end of the tube in Figure 7 is from the gas pressure generated by burning only on the end of the compacted bed being balanced by the decreasing bed volume. Reduced pressurization in the ignitor region delayed the onset of compressive reaction



**FIGURE 8. DDT mechanism as described in reference 4.**

and then SDT, even though heating LX-04 to 150°C somewhat increases<sup>5</sup> shock sensitivity. While SG records from the 70 %TMD test at 190°C are not available for assessing the early stages of DDT, the shock sensitivity at this temperature is considerably increased<sup>5</sup> to that of PETN. This increased shock sensitivity helped compensate for reduced convective burning, but  $l$  was still greater than at ambient temperature.

The small fragments from the middle region of the high-confinement tests that underwent DDT were from retonation in the compacted bed. The absence of the characteristic micro-cracking on the inner tube wall in this region may have been due to a gas layer between the bed and wall from the earlier reaction. The larger fragments beyond  $l$  are from detonation of the bed at the original loading density. At the ignitor end of the tube, the onset for small fragments corresponds to the displaced end of the compacted bed. Assuming compaction to TMD, that boundary is at  $l * \%TMD/100$ , in which %TMD is the initial value at loading. Retonation is not common in DDT, but it occurred<sup>4</sup> for 90/10 HMX/Al at 45.4 %TMD in a plastic tube. Retonation increases the fragment hazards in an accident scenario, as illustrated in Figure 4.

A somewhat related study<sup>6</sup> in steel tubes with 16.3-mm inner diameter, 50.8-mm outer diameter, and strong end closures was performed on ~115  $\mu$ m and Class A (~200  $\mu$ m) HMX mixed with various percentages of wax. These tests were at ambient temperature. For an 88/12 mixture at 69.3 %TMD,  $l$  was 273 mm for the ~115  $\mu$ m HMX and DDT failed for the Class A HMX. For a 91/9 mixture at 69.9 %TMD,  $l$  was 143 mm for the ~115  $\mu$ m HMX and 210 mm for the Class A HMX. The latter results are more characteristic of 70 %TMD LX-04 at ambient temperature. Because wax has a 1.00 g/cc density versus 1.85 g/cc for Viton, the 85/15 HMX/Viton mix in LX-04 has 84.7% volume of HMX, nearly the same as the 84.2% volume of HMX in 91/9 HMX/wax. It appears the DDT is based more on the %volume of the energetic ingredient in the mixture than its %weight as normally reported for the formulation.

The previously cited study had bed loadings of only ~70 %TMD. In another study having the same arrangement loaded with 91/9 RDX/wax,<sup>7</sup>  $l$  was 275 mm at 69.8 %TMD with a minimum of 120 mm at both 81.0 and 90.1 %TMD. Beds packed higher than 90 %TMD have limited connected

porosity for convective burning; and beds packed lower than 70 %TMD easily compact, which limits the initial pressure buildup by increasing the gas volume between the bed and ignitor. These data suggest that the minimum  $l$  for LX-04 will be between 70 and 90 %TMD.

## SUMMARY AND CONCLUSIONS

The tests to date have exhibited a wide range of reaction violence. In high confinement at ambient temperature, all porous beds detonated with the minimum  $l$  at 70 %TMD. While DDT failed at near TMD, the reaction is still violent enough to fragment the tube into many pieces. Heating the beds to just below the phase transition in HMX inhibited DDT, so much that it failed for a bed at pour density; 51.3 % TMD. Heating to 190 °C, just above the phase transition in HMX, still somewhat inhibited DDT based on longer  $l$  than at ambient temperature. Reactions in medium confinement for near TMD loadings were less violent at both ambient temperature and 186°C. The effects of heating are to inhibit convective burning by making porous beds easier to compact, partly offset by increased shock sensitivity. In high confinement, the ambient temperature data appear most similar to that for 91/9 HMX/wax, which has nearly the same %volume of HMX as LX-04.

## ACKNOWLEDGEMENTS

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